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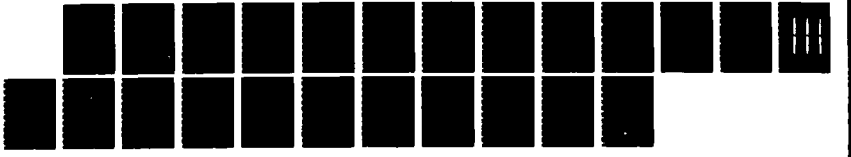

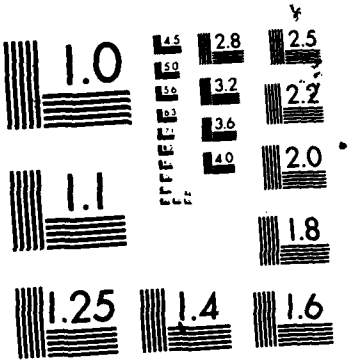

**Naval Research Laboratory**

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# USAF/NRL Surface Acoustic Wave Vapor Sensor Program

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19 ABSTRACT (Continue on reverse if necessary and identify by block number)

A sensitive, inexpensive chemical microsensor is described that is capable of meeting performance requirements for DoD applications. Experimentally obtained frequency shifts for known mass loading compare favorably with theoretically predicted mass sensitivities for the 31, 52 and 112 MHz surface acoustic wave (SAW) devices. The 300 MHz SAW device exhibits a frequency stability of better than 1 part in $10^7$, as well as good temperature and pressure stability under ambient conditions. Response times were generally less than 10 sec. When coated with a fluorepoxy pre-polymer this device exhibited a calculated detection limit of 0.04 mg/m$^3$ for DMMP.

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# USAF/NRL SURFACE ACOUSTIC WAVE VAPOR SENSOR PROGRAM

## BACKGROUND

The detection and identification of hazardous chemicals is an important problem. Numerous technologies presently exist for monitoring chemical threats but few are capable of meeting the extremely difficult performance requirements of the Department of Defense. These requirements include small size, ruggedness, automatic operation, sensitivity, selectivity, long shelf life, and minimal maintenance.

Recent advances in microelectronic technology have spawned a new generation of sensor devices all based on planar microfabrication techniques. These sensors are small "chip" size devices which have many of the characteristic features (e.g. manufactuarable, solid state, electronic output) that have been used to great advantage by the microelectronics community. Chemical microsensors have emerged which offer great potential for monitoring hazardous materials. Among these are CHEMFETs, Microdielectrometers, MIS diodes, Chemiresistors, and Surface Acoustic Wave (SAW) devices. All of these devices require a chemically selective coating to permit the detection of the chemical agent of interest. At the beginning of this USAF/AMD sponsored R&D program, the only chemical microsensor technology which was known to possess (at least theoretically) the required sensitivity for detecting war gases at or near miosis levels was the SAW device. Furthermore, several years of work had been spent (with U.S. Army CRDC support) developing coatings for this device which offered selectivity to organophosphorus compounds typically encountered in nerve gas formulations. As a result, the SAW device was selected as the most promising candidate microsensor for development into a DOD suitable chemical agent monitor.

The emphasis of the USAF/AMD program at NRL has been on the engineering of very high frequency (i.e. 300 MHz) SAW sensor devices and hybrid microelectronic support electronics. It was felt that the development of such a system would accomplish several objectives in a fashion which was complementary to other SAW research programs being

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conducted by NRL with U.S. Army funding. First, the system would allow the lead time for engineering of a complete prototype instrument to be shortened since the engineering development work on the USAF SAW sensor subsystem could be conducted in parrallel with more fundamental U.S. Army coating development work. In this way, advanced sensor devices would be available at or near the same time that optimized selective coatings were available. Second, fabrication of a breadboard 300 MHz sensor device would allow potential problems to be identified early in the program. This approach would allow a proof of concept demonstration prior to initiation of a major development program.

The principle by which a SAW vapor sensor operates is conceptually quite simple. The surface acoustic Rayleigh wave is generated on a very small polished slab of a piezolelectric material (e.g. quartz) with an interdigital electrode array which is lithographically patterned on the surface at each end of the device. When the electrode is excited with a radio frequency voltage, a Rayleigh wave is generated which travels across the device surface until it is "received" by the electrode at the other end. The Rayleigh wave has most of its energy constrained to the surface of the device and thus interacts very strongly with any material which is in contact with the surface (e.g. a chemically absorbent coating). Changes in mass or mechanical modulus of the coating produce corresponding changes in the Rayleigh wave velocity. The most common configuration for a SAW vapor sensor is that of a delay line oscillator in which the RF voltage output of one electrode is amplified and fed into the other electrode. In this way the device resonates at a frequency determined by the Rayleigh wave velocity and the electrode spacing. If the mass of the chemically selective coating is altered, then this changes the wave velocity which is measured as a shift in resonant frequency of the device.

The SAW vapor sensor is clearly quite similar to bulk wave piezoelectric quartz crystal sensor such as that originally described by King (1) and subsequently investigated extensively by Guilbault et.al. (2). Both devices respond to mass changes of coating deposited on the device surface. However, SAW devices possess several distinct advantages including substantially higher sensitivity (owing to the much greater device operating frequencies which are possible with SAW), smaller size, greater ease of coating, and improved ruggedness. Direct comparisons of SAW and bulk wave quartz crystal vapor sensors have been conducted by Vetelino et. al. who observed significantly larger signals using SAW devices (3). Other workers have reported on the mechanism of SAW vapor sensor response and model coating studies (4,5).

## THEORETICAL SCALING LAWS FOR SAW VAPOR SENSORS

The signal provided by a SAW oscillator vapor sensor can be described by the following theoretical equation (4):

$$\Delta f = (k_1 + k_2) f_0^2 \, h \rho' - k_2 f_0^2 \, h \left( \frac{4\mu'}{v_r^2} \left( \frac{\lambda' + \mu'}{\lambda' + 2\mu'} \right) \right) \quad (1)$$

where $\Delta f$ is the SAW oscillator frequency change produced by the vapor absorbed into the coating, $k_1$ and $k_2$ are material constants for the piezoelectric substrate, $f_0$ is the unperturbed resonant frequency of the SAW oscillator, h is the coating thickness, $\rho'$ is the coating density, $\mu'$ is the shear modulus and $\lambda'$ is the Lame` constant of the coating, and $V_r$ is the Rayleigh wave velocity in the piezoelectric substrate (3159 m/sec for Y-X Quartz). This relationship assumes that the SAW device coating is isotropic and non-piezoelectric. Furthermore, the relationship is valid only for very thin films (e.g. less than 0.2% of the acoustic wavelength thick). For thicker films, equation 1 can only provide estimates of the signal magnitude. When organic coatings are employed, it is often found that the second term of equation 1 is negligible because the shear elastic modulus of the coating ( $\mu'$) is small compared to the square of the Rayleigh wave velocity ($V_r$). Under these conditions, equation 1 reduces to:

$$\Delta f = (k_1 + k_2) f_0^2 \, h \rho' \quad (2)$$

For Y-X Quartz SAW devices, $k_1 = -9.33 \times 10^{-8}$ $m^2 \cdot sec/kg$ and $k_2 = -4.16 \times 10^{-8}$ $m^2 \cdot sec/kg$. The product of the coating thickness (h) and its density ($\rho'$) is the mass per unit area on the device surface. Equation 2 predicts that the signal obtained from a given mass loading (h $\rho'$ product) will increase with the square of the operating frequency of the SAW oscillator. Furthermore, operating frequency determines the size of the device since it imposes size requirements on the interdigital electrodes used to generate the Rayleigh surface wave. As the operating frequency increases, the device area (and cost) decreases. Higher operating frequencies permit thinner coatings to be employed with a corresponding improvement in response time since vapor diffusion into the coating will be quicker.

Higher operating frequencies also result in greater baseline noise which hinders detection at the lowest concentrations. All of these considerations result in a set of scaling laws (detailed in reference 6) which can offer guidance in predicting the ultimate performance capabilities of SAW vapor sensor technology. The key assumption in these predictions is that the sensitivity increases with the square of the frequency. Preliminary studies indicated that this theoretical model was valid (4). Nevertheless, additional studies were performed to provide additional substantiating evidence of the model's accuracy.

## Experimental Investigation of Scaling Behavior

In order to verify the preceeding theoretical model a series of experiments were conducted on devices operating at frequencies of 31, 52, and 112 MHz. In these experiments, the frequency shift produced by different mass loadings applied to the SAW oscillator was measured. A highly reproducible mass loading on each device was obtained from coatings of various thicknesses deposited by the Langmuir- Blodgett (L-B) technique. Devices designed for 300 MHz operation were not included in the study because the device package made it impossible to coat these devices using the Langmuir-Blodgett technique. Coatings were applied to the devices and then connected to the RF electronics in order to measure the resonant frequency of the coated device. Then, with the device still connected to the electronics, the coating was removed using a Q-tip soaked with solvent for the coating. When the device was clean and dry, the resonant frequency was again measured. The difference between the clean and coated resonant frequencies was recorded as the frequency shift produced by the coating. It was expected that the same number of layers of coating would produce correspondingly greater frequency shifts as the device frequency was increased from 31 to 52 and then 112 MHz. The results of this investigation are presented in figure 1. The strong dependence of sensitivity (defined as the slope of the frequency shift vs number of layers) is clearly evident. The theoretical model would predict that these slopes should increase in the ratio of 1 : 2.8 : 12.8 as the measurement frequency is increased from 31 to 52 to 112 MHz. The experimentally measured slope increase in the ratio of 1 : 5 : 19 which suggests that the sensitivity increases even more with frequency than one would anticipated from the theory.

In addition to improved sensitivity, higher operating frequencies also afford faster response times since thinner coatings are employed. A 31 MHz and 112 MHz SAW device were coated with the same material (Poly(ethylene maleate)) to a thickness which provided similar sensitivities to DMMP vapor. When exposed to 2 ppm of DMMP, the 31 MHz device required more than 1000 seconds to reach equilibrium. The 112 MHz device required less than 100 seconds. Preliminary results with a 300 MHz SAW device, having a different coating than that used in the 31 and 112 MHz studies, have revealed response times of less than 10 seconds. In fact, reponse times this small are probably determined by the speed with which the challenge gas mixture can be made rather than by the

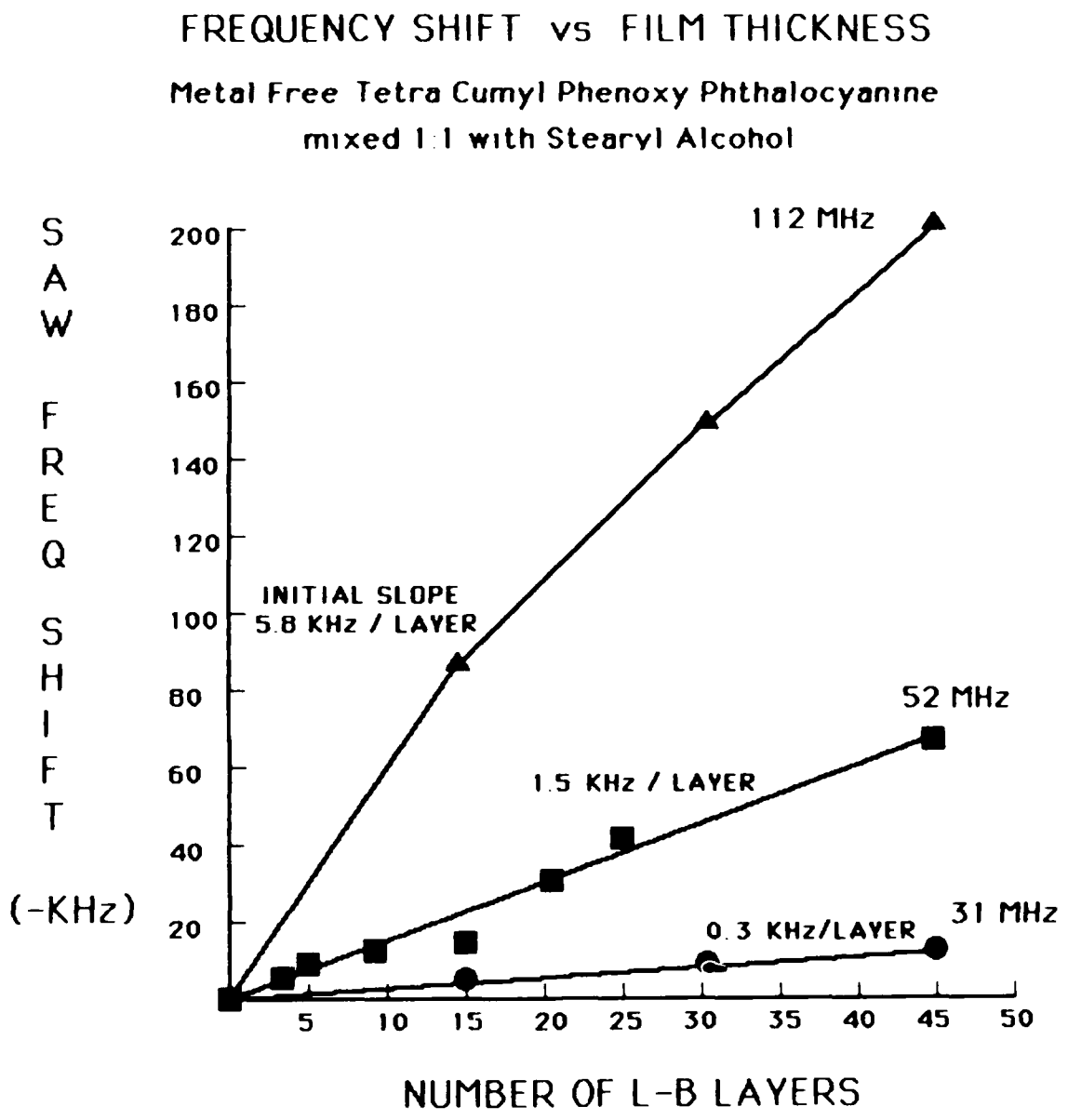



Fig. 1 — Experimental frequency shifts obtained from various mass loadings measured with 31, 52, and 112 MHz SAW oscillators

response time of the sensor itself. If the response time is limited solely by the rate of diffusion into the coating, then one expects to observe an inverse fourth power dependence of reponse time on operating frequency. This is due to the fact that the time for diffusion to occur is related to the inverse square of the coating thickness and the coating thickness required to provide a given vapor sensitivity is related to the inverse square of the operating frequency. In practice this inverse fourth power dependence is not observed, probably because other factors besides simple diffusion into a coating are involved. Nevertheless, substantial improvements in response times are observed as the operating frequency is increased.

## 300 MHz SAW Vapor Sensor System Description

The objective of this work was to design the highest frequency SAW vapor sensor ever studied. A frequency of 300 MHz was selected as a suitable target frequency since it was possible to fabricate SAW devices at this frequency using optical lithographic tools. Devices at significantly higher frequencies (e.g. 600 MHz and above) usually require more exotic fabrication technologies such as X-ray or E-beam lithography. Theory indicated that a 300 MHz device would offer substantial improvements in sensitivity, size, and response time over previous SAW vapor sensor devices.

A dual SAW delay line oscillator configuration was chosen for the vapor sensor. In this design, two SAW delay lines are fabricated on the same substrate. One delay line is coated with the chemically selective film and the other is left uncoated. The frequencies of the two delay line oscillators are mixed to provide a frequency equal to the difference of the two oscillator frequencies. In this way, frequency drifts caused by ambient temperature and pressure fluctuations experienced by the SAW device are compensated. Furthermore, the difference frequency is much lower (e.g. several hundred KHz) than the frequency of the oscillators themselves (i.e. 300 MHz). This makes it much easier to measure the SAW vapor response using inexpensive, digital counter circuitry. The actual design of the 300 MHz dual SAW vapor sensor device is illustrated in figure 2. The "chip" is fabricated on ST-Quartz using gold metallization on top of a thin adhesion layer of titanium. The electrode configuration was selected to provide high resonant Q and minimal insertion loss from the coating. Overall size of the chip is 240 X 130 mils (6 X 3.3 mm). The active area of each SAW delay line is approximately 2 square millimeters. The SAW device is mounted in a conventional microelectronic 8 pin gold flat package whose internal volume is less than 60 microliters. The device is held in the package using epoxy and electrical connections are wire bonded from the device to the package connecting leads. The SAW delay line was fabricated to NRL specifications by SAWTEK, Inc. Orlando, Florida (P/N 851210). The conventional gold lid of the device package was replaced with a piece of Lucite fitted with two, 1/16 inch stainless steel vapor inlet and outlet tubes. This lid was held onto the top of the device using a small C-clamp.

The high frequency of this sensor required a new approach toward the fabrication of not only the SAW device but also the supporting electronics.

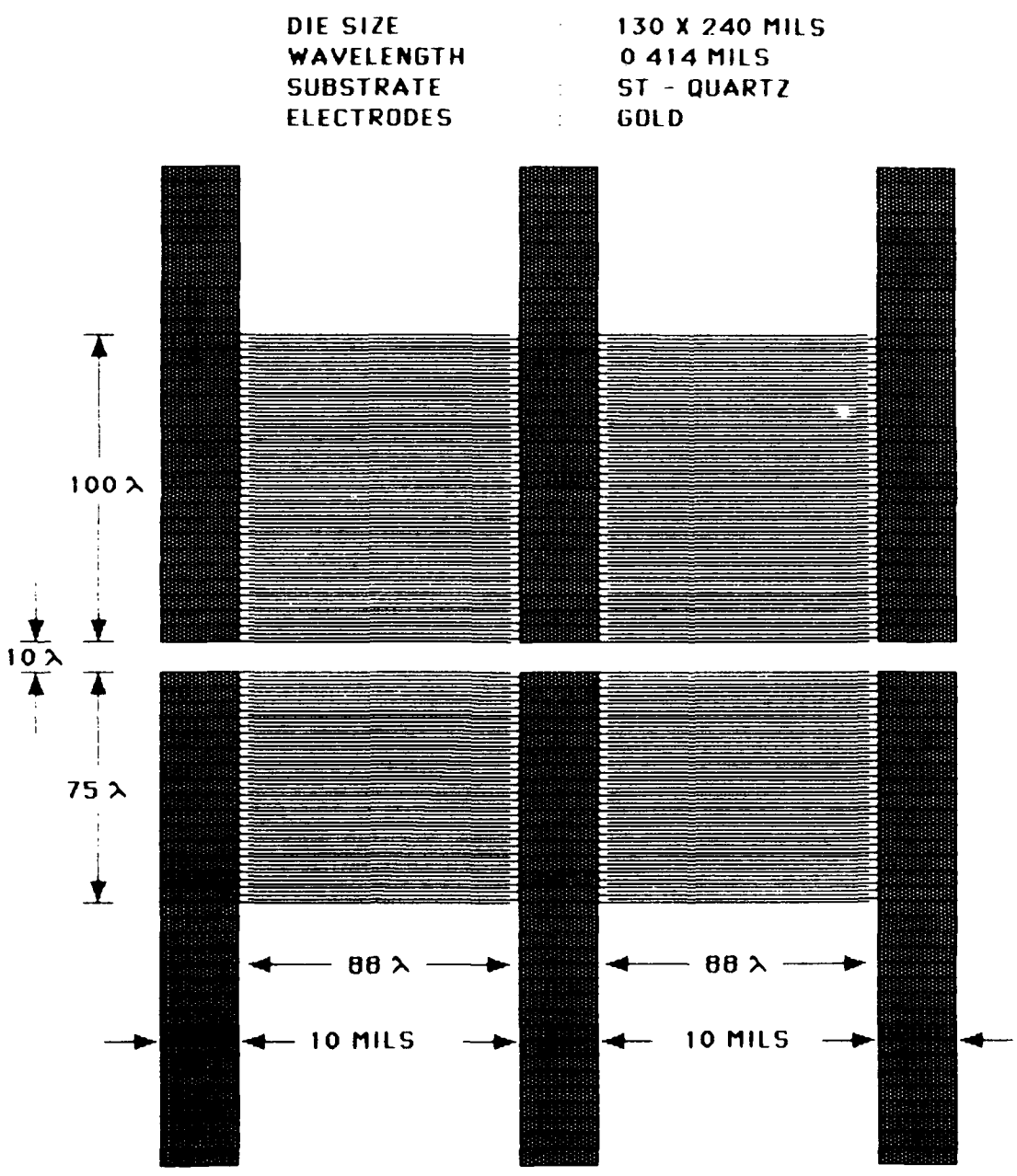



Fig. 2 — NRL 300 MHz dual SAW device

In order to improve stability and reliability and to reduce the size of the supporting electronics, hybrid fabrication technology was employed. The two RF amplifiers required to power the dual SAW delay line oscillators were contained in a 24 pin dual inline package. Each RF amplifier was a simple single transistor, tuned circuit design. Chip resistors, capacitors and transistors were used along with novel chip inductors microfabricated on glass. The final design consumed less than 500 mW of power and provided more than 20 db of gain at 300 MHz. The RF module can be reconfigured (i.e. tuned) to operate at other frequencies besides 300 MHz. The Dual 300 MHz SAW RF Module was fabricated to NRL specifications by SAWTEK, Inc. Orlando, Florida (P/N 852008).

The dual SAW device and RF module were connected together on a small printed circuit board to form a complete vapor sensor subsystem as shown in figure 3. Electrical power and output signal connections are made to this P.C. board. In addition, the mixer used in this device is mounted on the board external to the RF module. The mixer is a passive device (Mini-Circuits Lab, double balanced mixer, TFM-2) which could be incorporated into the RF module in future designs. The total volume occupied by the sensor system was less than 1 cubic inch.

The electrical performance of the complete dual SAW vapor sensor module proved to be quite acceptable. Power consumption (less than 1/2 Watt) was substantially below the 1 Watt design goal. The actual, uncoated, resonant frequency of the device was 290 MHz. A frequency stability of 1 part in $10^7$ measured over a 1 second interval was desired. The actual system (uncoated) was stable to better than 9 parts in $10^8$. This translates into a baseline noise level of less than 30 Hertz. Significant improvements in the stability of the oscillator may be possible through redesign of the SAW device. The present design utilized a common ground bus for the two delay lines. This common ground was found to cause some cross talk between the two oscillators thereby degrading the frequency stability.

The stability of the system to pressure fluctuations appeared to be good. Increasing the pressure on the device by about 0.1 atmospheres produced frequency variations of less than 4 KHz. Temperature stability of the system under normal room temperature fluctuations was very good. However, when a more aggressive temperature test was made (i.e. a 600 degree heat gun placed 3 feet away which made the system too hot to touch), large frequency changes (e.g. 100 KHz) were observed. While it was not possible in a quick test to determine unambiguously the source of the

drift, it appeared that the RF module was the most likely culprit. Small phase shifts caused by changes in temperature of capacitors or inductors could easily explain the observed temperature sensitivity. More attention to this problem will be required in future designs which must operate over the full MIL-SPEC temperature range.

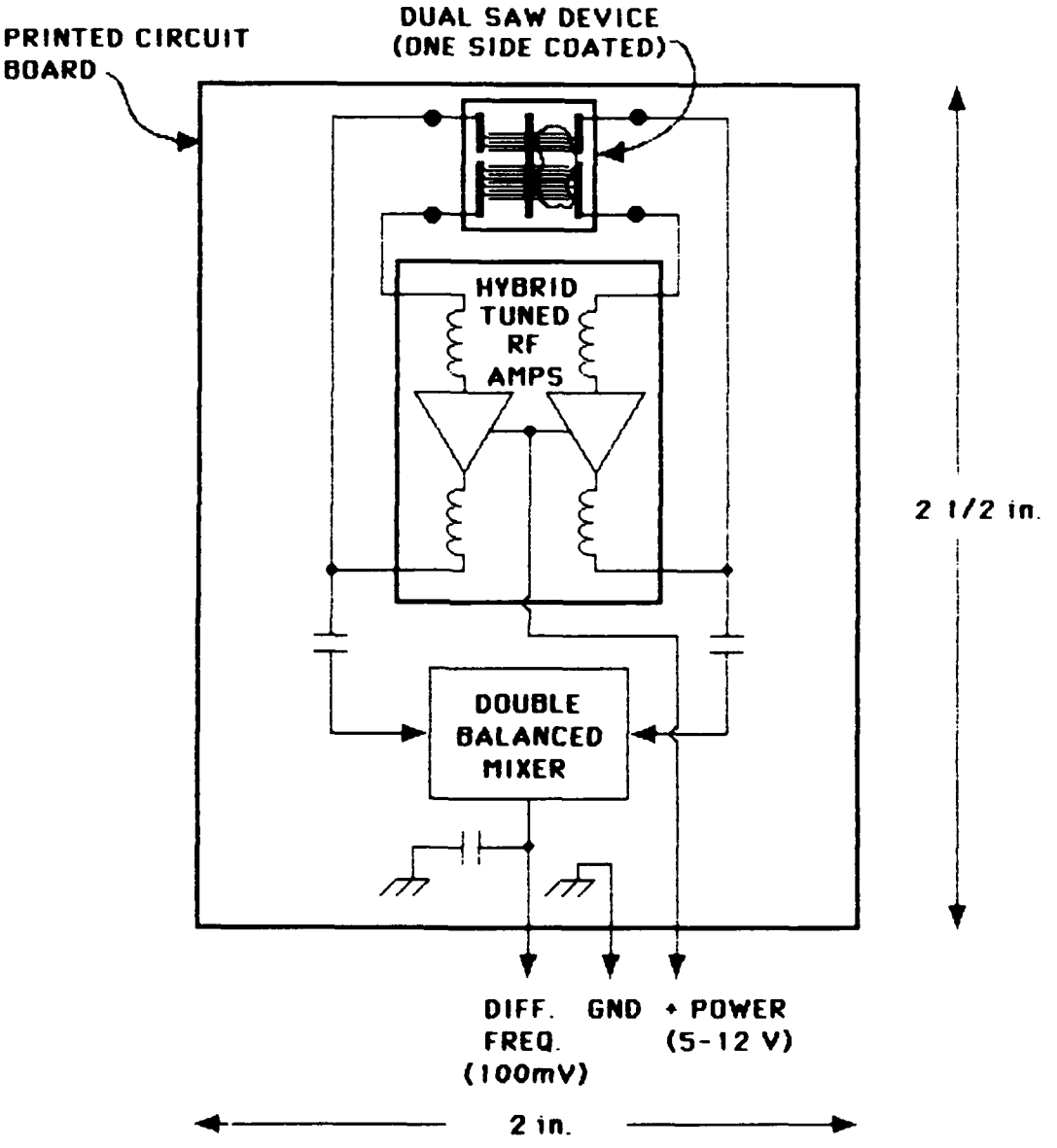
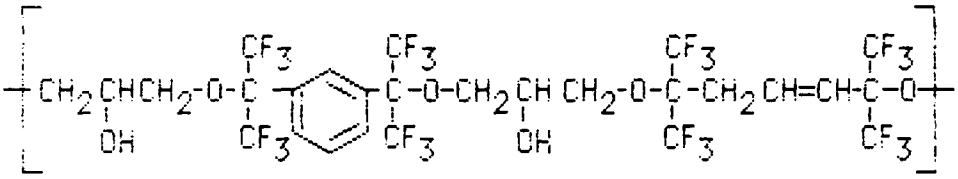



Fig. 3 — NRL 300 MHz dual SAW vapor sensor system

## SAW Coating Procedure

Results of SAW vapor sensor tests conducted at 31 and 112 MHz revealed that one of the best coatings for detecting G-agent simulants (such as DMMP) was a fluoroepoxy pre-polymer provided by Dr. Jim Griffith of NRL's polymer materials branch. This compound, dubbed "fluoro polyol" has been demonstrated to detect GB and GD and was used to evaluated the response of the 300 MHz SAW vapor sensor. It is an oligomeric material which is soluble in a number of organic solvents including chloroform. The structure of the material is shown below:



NRL's "Fluoro polyol"

The technique selected to deposit this material onto the SAW device was air brushing. Other techniques such as Langmuir-Blodgett dipping or spin casting could not be employed because the devices were packaged and wire bonded. This would make application of a uniform coating impossible. The only other method available was solvent evaporation in which a small drop of a dilute solution of coating in a volatile solvent is deposited onto the device surface and allowed to dry. This technique requires great care since the active area to be coated was only about 2 square millimeters in size.

Coating application was accomplished by spraying through a small mask positioned over the active area of the delay line to be coated. A dilute solution of approximately 0.1% by weight of fluoro polyol in chloroform was used. Compressed air was used as the propellant. The spray was adjusted to provide a barely visible deposit on a clean glass plate when sprayed from a distance of 3 inches for 15 seconds. This spray was then used to coat the device from a distance of 6 inches while the difference frequency was monitored with an oscilloscope and frequency counter. Mulyiple short bursts of the spray onto the device surface resulted in a coating which produced a total frequency shift of 260 KHz. The device was allowed to sit in clean, dry air for about 12 hours prior to testing.

## Experimental Investigation of Vapor Response

Exposure of the fluoro polyol coated SAW device to simulant and interferent vapor challenges was performed using a fully automatic vapor generating system designed and built at NRL. The system is capable of generating low concentrations of vapor (e.g. 0.1 - 10 ppm simulants) using thermostatted, gravimetrically calibrated, permeation tubes. Higher vapor concentrations (e.g. 100 - 10,000 ppm interferences) can be generated using gravimetrically calibrated bubblers. Vapor streams from both the bubblers and permeation tubes can be diluted using precise, computer operated, mass flow controllers. Dry air is used as a carrier gas and the outlet of the system is at ambient pressure. All tubing in the system is stainless steel to minimize corrosion and wall adsorbtion. The flow rate of vapor presented to the sensor is computer controlled and programmable. Vapor generation and acquisition of sensor response data is performed with an APPLE IIe computer. The SAW sensor signal from the RF module is fed into a Systron-Donner frequency counter which communicates to the computer via an IEEE 488 interface bus.

Four low concentration vapors (ammonia, methane sulfonyl fluoride, dimethyl acetamide, and dimethyl methyl phosphonate; generated using permeation tubes) and eight high concentration vapors (dichloro ethane, water, toluene, isooctane, diethyl sulfide, tributyl phosphate, 2-butanone, and 1-butanol; generated using bubblers) were used to test the SAW sensor. The sensor was exposed repeatedly to clean air carrier and then to air contaminated with a particular vapor. The difference between the SAW frequencies measured during clean air and contaminated air exposure was used to determine the magnitude of sensor response. Data were obtained at four different concentrations for each of the vapors investigated.

A summary of the vapor exposure data is illustrated graphically in figures 4 and 5. The responses are expressed in Hertz per part per million of vapor concentration. The results show a very high degree of selectivity for the simulants (i.e. DMMP, DMAC, and MSF) as opposed to the interferents. The largest interferent (besides ammonia) is tributyl phosphate which is the interferent most closely related to DMMP. However, the fluoropolyol is still more than 150 times more sensitive to DMMP than to tributyl phosphate. The selectivity against water vapor is really excellent with the fluoro polyol responding more than 20,000 times more strongly to DMMP than to water.

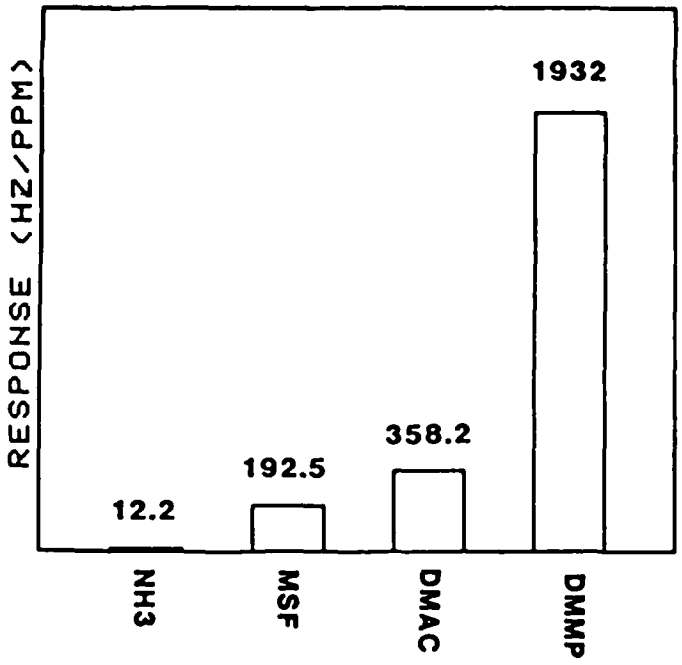



Fig. 4 — Summary of SAW responses to simulant vapors

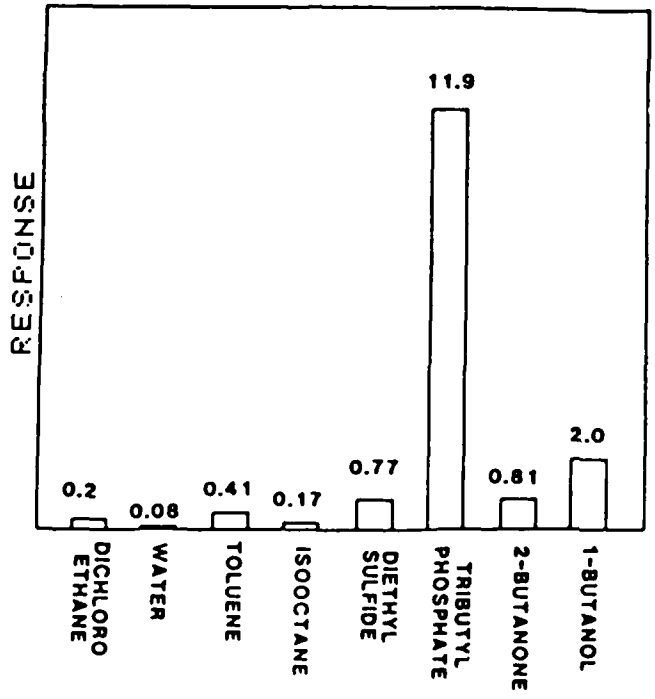



Fig. 5 — Summary of SAW responses to interference vapors

Figure 6 shows a typical response curve obtained from an exposure to 0.5 ppm (about 2 $mg/m^3$ ) of DMMP. The results are very good. Response time is less than 10 seconds and is probably limited by the ability of the vapor generator to switch vapor streams and establish equilibrium. At this concentration, the sensor is providing a signal of more than 5 KHz with a baseline noise level of less than 30 Hz. This is a signal to noise ratio of more than 150 to 1. A conservative estimate of the detection limit based on these results is 0.04 $mg/m^3$ assuming a detection threshold signal to noise ratio of 3 to 1. These results represent the first attempt at vapor detection using the 300 MHz SAW sensor. Further improvements to the system which reduce the noise or increase the signal will yield still lower detection limits.

A calibration curve for DMMP is shown in figure 7. Calibration curves for other vapors tested and raw data from the vapor exposure experiments is contained in the Appendix.

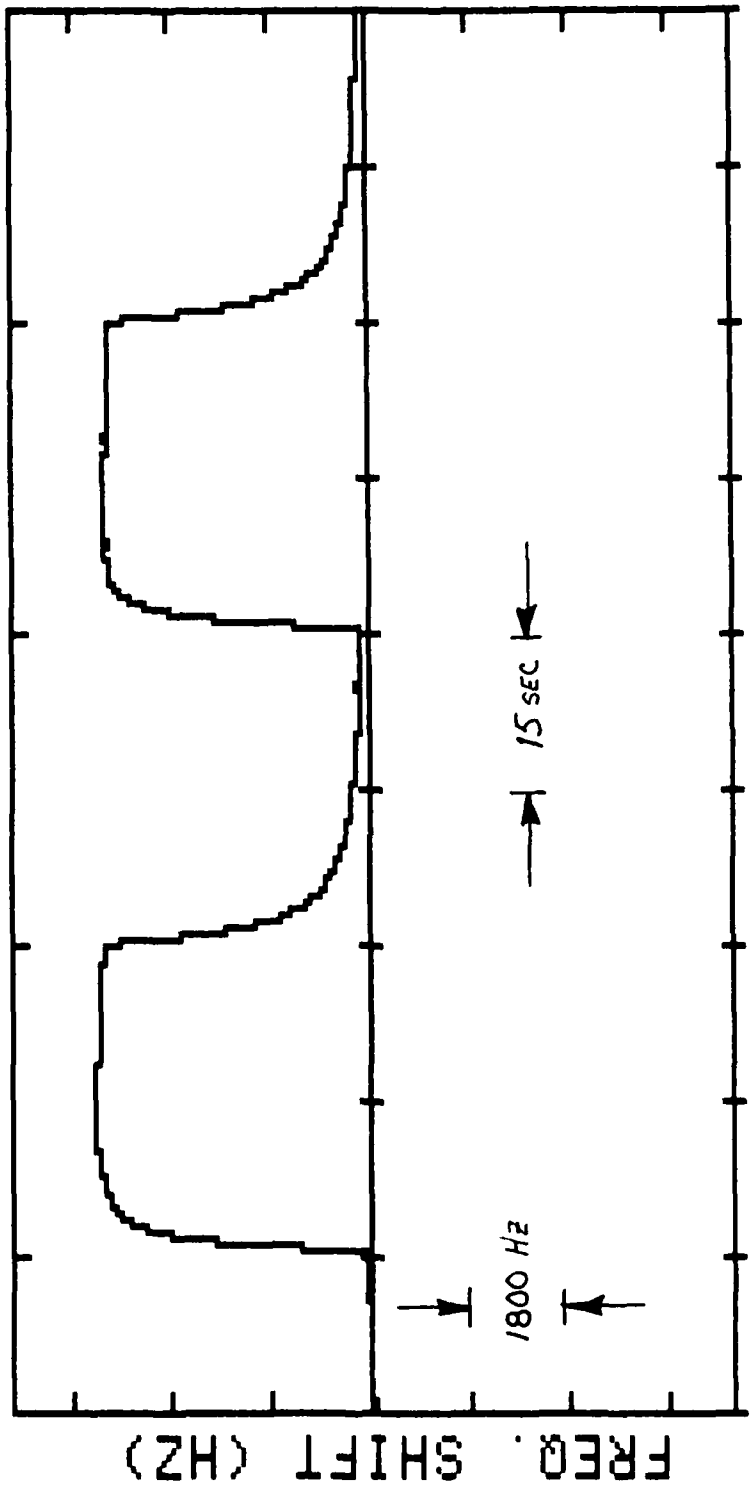



Fig. 6 – Typical response of a fluoro polyol coated 300 MHz SAW device exposed to consecutive pulses of 0.5 ppm DMMP in air

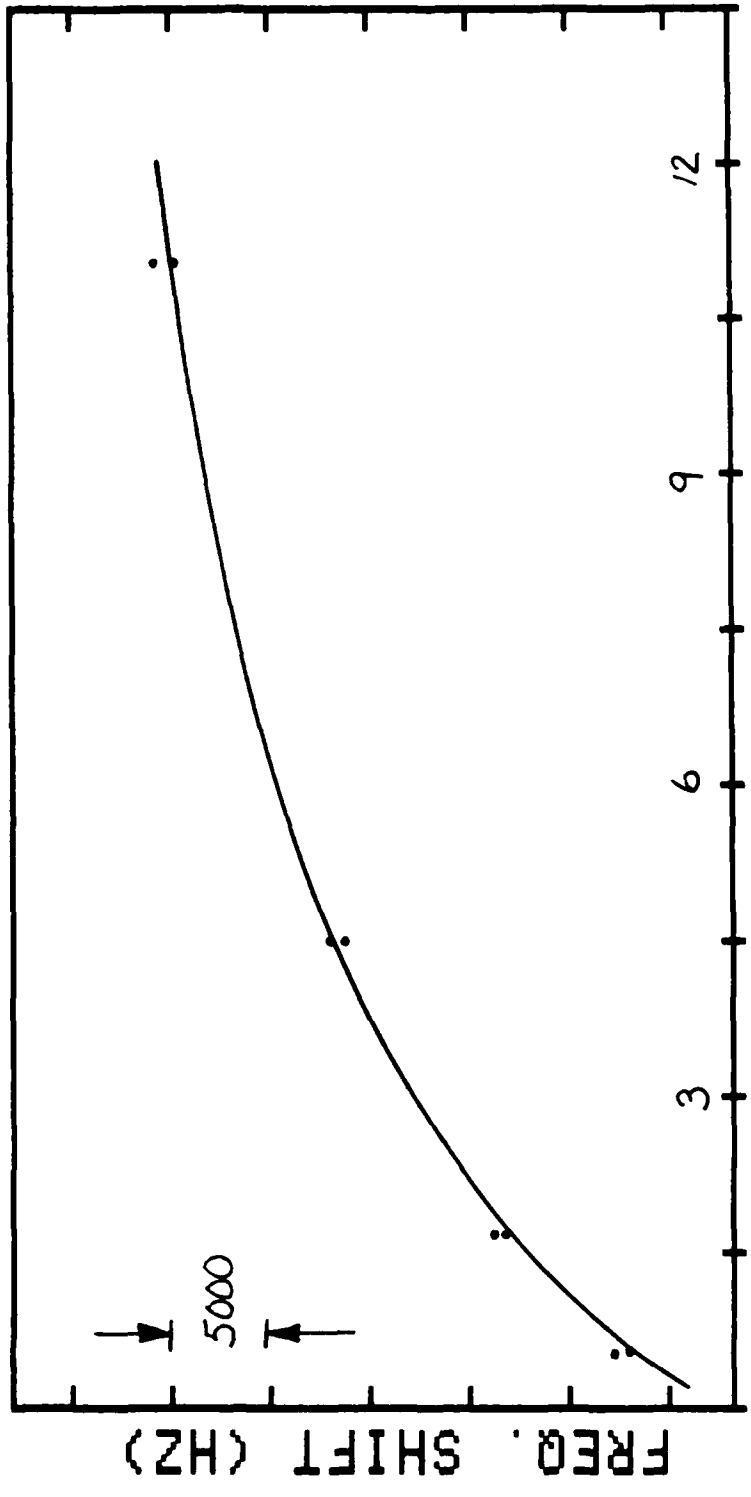



Fig. 7 — Calibration curve for fluoro polyol coated 300 MHz SAW device exposed to various concentrations of DMMP in air

## CONCLUSION

The experiments described here have demonstrated the validity of a theoretical response model developed at NRL which predicts a SAW device sensitivity which increases with the square of the frequency.

A 290 MHz dual SAW delay line oscillator and a hybrid RF electronics module has been designed, fabricated, and tested. The electrical performance of the device met or exceeded the design targets. Further attention should be paid to the areas of baseline noise reduction, temperature drift compensation of the RF electronics, and improved packaging of the SAW device.

An oligomeric fluorinated pre-polymer material developed at NRL, "fluoro polyol" was selected as the coating to be used in evaluating the SAW vapor sensor response. This compound has been shown to detect GB and GD with slightly less sensitivity than that measured with DMMP. The material is soluble in chloroform and was applied to the active area of the SAW device using a spraying technique.

The vapor responses measured during the preliminary study were very good. Response times were usually less than 10 seconds and it is believed that this time is determined not by the SAW sensor, but by the vapor generation apparatus. The magnitude of the baseline noise fell within the predicted limits as did the magnitude of the vapor responses. Strongest responses were obtained when the sensor was exposed to DMMP. A detection limit of less than 0.04 $mg/m^3$ is estimated for the present device. Very modest improvements in coating technology (i.e. materials and methods of application) along with modest reductions in baseline noise should permit the selective detection of organophosphorus compounds at concentrations below 0.01 $mg/m^3$.

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